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Low Energy Pre-blended Mortars: Part 2 – Production and characterisation of mortars using a novel lime drying technique

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Abstract

The presence of free water in mortars destined for silo or bagged storage can lead to the degradation of the binder phase. Such water may be present as a result of using wet, as-delivered sand or as a consequence of prior processes such as de-activation of Roman cement. Thus, water must be removed from the system prior to storage. Part 1 of this paper describes the control of a technique by which quicklime is added to the wet system which principally dries it by both slaking the quicklime and evaporation as a consequence of the exothermic slaking reaction. Two examples of mortars are presented in which excess water is removed from the system by the inclusion of quicklime. In the first, the water is present in the as-delivered sand and the binder is a combination of the slaked lime and ggbs. In the second, the water remains after pre-hydration of a Roman cement which is a process to retard its rapid setting

characteristics. It is shown that optimally dried mortars are not subject to degradation following storage of both mortar types.

Keywords: mortar, sand drying, ggbs, slaked lime, storage, Roman cement

1 Introduction

The first part of this paper [1] described a technique by which the addition of quicklime to wet sand could be controlled to produce Formulated Lime mortars which are dry and suitable for long-term storage in silos or bags prior to final wet mortar production. The principal process-control factors to achieve optimal drying have been identified as lime addition based upon a fraction of the stoichiometric requirement for complete slaking of the quicklime (i.e. 3.113 g of CaO being required to combine with 1 g water), free moisture content of the sand and mixing time of the combined sand and quicklime blend followed by a 24 hour “rest period”. The philosophy can be equally applied to other factory produced mortars in which free water may remain following the formulation process.

A fundamental feature of this concept is that water which is not contained within the pore structure of the sand, i.e. free water, rather than the total water content is the one which must be controlled. In this scenario the datum state of the aggregate is its saturated surface dry (SSD) condition in which the sand pores are completely filled whilst the free moisture is zero. Trials of mortars produced with natural hydraulic lime (NHL 3.5) showed that mortars in which the binder was mixed with SSD sand and stored for 8

weeks in a sealed box exhibited no degradation when compared to companion mortars which were immediately produced without the storage period.

This part of the paper describes the application of the technique in the production of two types of mortar. The first are Formulated Lime mortars, M1 and M5, produced with the addition of ggbs as the hydraulic phase. Formulated Limes are specified in BS EN 459-1:2010 [2] as being limes with hydraulic properties and comprising lime and added material with hydraulic and/or pozzolanic properties whilst the strength classes M1 and M5 (compressive strengths of 1 MPa and 5 MPa respectively) are specified in BS EN 998-2:2010 [3]. The mix design process has to ensure that the quicklime addition is sufficient to yield both the dry mortar for storage and also the correct amount of ggbs, principally the ggbs/slaked lime (CH) ratio, to yield the desired mortar type whilst maintaining the required volumetric proportions of binder to aggregate. The properties of mortars made immediately after addition of the hydraulic component are compared to those in which the dry blended mortar was stored for 10 weeks prior to mortar production. The second application is in the production of De-Activated Roman Cement (DARC) mortars suitable for long-term storage. A characteristic of Roman cements is their rapid setting such that retardation is required for the production of practical mortars. A workable life of 1 – 2 hours has been specified for render mortars [4] for which a pre-hydration process has been developed [5]. In essence, this process involves adding an initial controlled amount of water to the Roman cement such that sufficient reaction takes place to retard the setting of the mortar when the fresh mortar is subsequently manufactured. During that study it was observed that DARC mortars stored for 1 – 12 weeks exhibited some degradation during storage, thought to be due to the presence of residual water following the de-activation process. Thus, the situation is akin to that of the presence of excess water in the Formulated Lime mortars which needs to be

removed, although in this case the water results from a factory-based process rather than from as-delivered materials. Mortars with the addition of quicklime following the de-activation stage are compared with samples which were oven-dried at this point as well as control samples.

Part A: Formulated Lime Mortars

2 Materials and Methods

2.1 Materials

A siliceous sharp sand was used throughout and was oven dried to constant weight at 110^{o}C prior to use. Two different quicklimes, C1 and E, were used for the preparation of the lime-dried mortars. The chemical and physical properties of these materials, together with properties of the slaked limes, were reported in Part I of this study [1]; Table 1 summarises the key properties of the slaked limes and a commercial CL90 which has been used as a control. ggbs was supplied by Civil & Marine Slag Cement from the Frodingham plant, UK.

Lime	C1	E	CL90
Free lime (% CaO)	70.9	68.7	71.3
Insolubles (wt%)	5.8	4.8	3.8
CaO (wt%)	0.3	0	0.9
$Ca(OH)_2$ (wt%)	93.2	90.8	93.0
$CaCO_3$ (wt%)	2.9	6.9	2.3
A_{BET} (m^2/g)	15.19	18.16	11.26
V_{TOT} (cm^3/g)	0.098	0.109	0.072
Bulk Density (kg/m^3)	585	559	575

Table 1: Properties of limes

2.2 Mortar Production

2.2.1 Mix design of lime-dried mortars

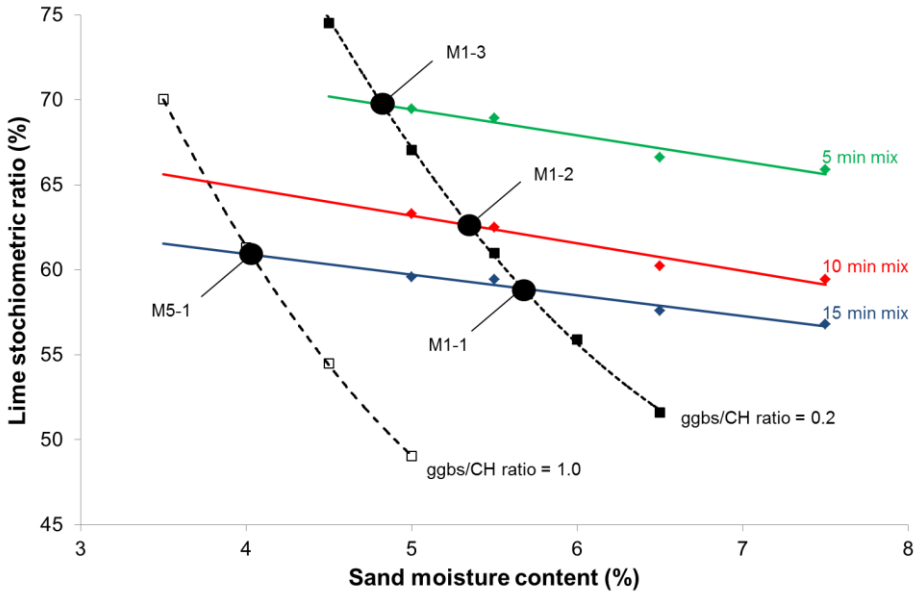


Fig 1: Example of mix design for optimum mortars using lime E.

Figure 5 of ref [1] shows that the optimum stoichiometric ratio for quicklime addition to dry the sand decreases with increases in either the sand moisture content or the mixing time of the wet sand and quicklime. This is represented for lime E in Figure 1 by the 3 linear relationships. The design of the lime-dried mortar formulations was intended to produce mortars of M1 and M5 classification according to BS EN 998-2 [3]. The approximate ggbs/CH ratio (by weight) required to achieve the stated classification was 0.2 and 1.0 respectively where the mortars were produced to a common flow of 170 mm [6]. Obviously, the precise ratio required to yield a specified strength for any mortar will also be related to the purity of the quicklime, grading of the sand as it affects workability, the sand density and the density of the $Ca(OH)_2$ (also referred to by its cement chemistry notation of CH) generated during the drying process as they affect volumetric mix proportions. Once a sand:binder ratio has been selected it is possible to calculate how much $Ca(OH)_2$ is required for a specified ggbs/CH ratio. Hence, knowing how much quicklime would be required to chemically combine with all of the free water for a given free moisture content of the sand, the stoichiometric ratio of quicklime addition may be calculated which generates the correct amount of $Ca(OH)_2$ for the specified ggbs/CH ratio, after accounting for the purity of the quicklime. For instance, for a particular mortar batch size and mix proportions, if 1381 g of $Ca(OH)_2$ is required to yield the desired ggbs/CH (equivalent to 1045 g of CaO), whilst theoretically requiring 1771 g of CaO to chemically combine with the free water in the sand, this would represent a stoichiometric ratio of 59%. In this procedure the density of the ggbs and the CH have been measured but no allowance was made of any interactions between the two phases which might affect their packing efficiency.

The associated values of stoichiometric ratio and sand free moisture content for both ggbs/CH ratios have been superimposed on Figure 1 and the intersection of the

relationships indicates the optimum drying conditions for each mortar; the 4 mortars using quicklime E detailed in Table 2 are indicated on Figure 1. The same approach was adopted for mortars using quicklime C1 In accordance with the terminology previously described [1] all mortars are considered optimally dried. All mortars were produced at a constant sand:binder ratio of 2.25:1 by volume.

Code	Lime type	Stoich ratio (%)	Sand m/c (%)	Mix time (min)	ggbs/CH ratio	w/b ratio
M1-1	E	58.87	5.69	15	0.2	1.35
M1-2	E	62.62	5.35	10	0.2	1.36
M1-3	E	69.72	4.81	5	0.2	1.36
M1-4	C1	61.20	5.71	15	0.2	1.37
M1-5	CL90	~	~	~	0.2	1.41
M5-1	E	60.9	4.03	15	1	1.03
M5-4	C1	61.2	4.08	15	1	1.05
M5-5	CL90		~	~	1	1.06

Table 2: Mix design

The experimental programme was designed to determine the effect of various parameters of the lime-drying process on the physical properties of the resulting hardened mortars. The formulations allow assessment of the influence of the drying process, the drying time and the quicklime source; mortars using CL90 provide control data.

2.2.2 Preparation of lime-dried mortars

143 Ten kg batches of sand were prepared containing known amounts of free water (wt.%
144 basis). The sand was then mixed in a Hobart mixer with appropriate quantities of
145 quicklime for the specified period of time (i.e. 5, 10 or 15 min). Following mixing, the
146 lime-dried materials were stored in sealed containers for 24 hours to ensure complete
147 hydration of the quicklime; this step was included after previous observations indicated
148 the presence of free water in the blend at the completion of the initial mixing [1]. The
149 required quantity of ggbs was then added in order to provide the desired mix
150 proportions. At this stage, the dry mortar mix was divided into two equal portions. One
151 portion was stored in a sealed plastic container and retained under laboratory conditions
152 (20^{o}C, 50% RH) for 10 weeks; the other portion of the mix was prepared immediately.
153 These mortars have been termed “stored” and “fresh” respectively.
154
155 **2.2.3 Production of plastic mortars**
156
157 Lime dried mortars were produced by adding mix water to the “dry mortar” whereas the
158 control mortars were produced by adding the water to a blend of CL90 and SSD sand.
159 All plastic mortars were produced in a Hobart mixer with a mixing time of 15 minutes.
160 Appropriate quantities of water were added to give a flow table value [7] of 170mm (+/-
161 5mm). This value was considered to provide materials with workability consistent with
162 that observed in typical construction practice [8]. The required free water:binder ratio to
163 achieve the specified flow value for each mortar is displayed in Table 2.
164
165 **2.2.4 Curing of mortars**
166
167 Following preparation, the plastic mortars were cast into steel moulds of the required
168 geometries (see section 2.3). The samples were initially cured within the moulds for 3

169 days at 20^{o}C and covered by a polythene sheet to minimise evaporation. The moulds
170 were then stripped and the samples cured for a further 4 days under the same
171 conditions. Unless stated otherwise, extended curing was then carried out at 20^{o}C (±
172 2^{o}C) and 65% RH (± 5%), hereafter referred to as air curing. The level of CO_2 in the
173 curing room was assumed to be the standard atmospheric concentration (0.033 ±
174 0.001% by volume). For comparison, some samples (for strength testing only) were
175 subject to extended curing under water, also at 20^{o}C.
176
177 **2.3 Analytical Methodology**
178
179 **2.3.1 Compressive and Flexural Strength**
180
181 The test specimens were prepared in steel moulds of 40 x 40 x 160 mm dimensions.
182 Mortar was placed in 2 layers and compacted using a vibration table. The three-point
183 flexural and compressive strengths of the hardened mortars were determined using an
184 Instron 4206. Crosshead speeds of 0.5 mm/min and 1 mm/min were used for flexural
185 and compressive testing respectively. Measurement of compressive strength was
186 carried out at 7, 28, 91 and 365 days. For air-cured samples, flexural strengths were
187 determined at 7 and 91 days whereas water-cured materials were tested at 28 and 91
188 days.
189
190 **2.3.2 Density and Porosity**
191
192 The bulk density and total open porosity of the hardened mortars were assessed in
193 accordance with BS EN 1015-10 [9] and BS EN 1936 [10] respectively (vacuum
194 saturation method). The reported values are the mean of three individual samples.

2.3.3 Sorptivity

The water sorptivity of the hardened mortars was determined according to the methodology described in detail by Hall [11]. The test specimens were prepared in 70 mm steel cubes and cured as described in section 2.2.4. Prior to testing, the samples were conditioned at 60^{o}C to constant weight in an atmosphere circulated over silica gel and soda lime. The sorptivity was measured through the moulded bottom surface; the vertical faces were sealed by the application of water-resistant tape. The reported values represent the mean of three individual samples.

2.3.4 Water Vapour Permeability

The water vapour permeability of the hardened mortars was determined using a modified version of the methodology described in BS EN 1015-19 [12]. The test specimens were cast in circular steel moulds, producing specimens of 180 mm diameter and 20 mm thickness. The mortar discs were then subjected to the curing regime specified in section 2.2.4. After curing, the samples were prepared for analysis at 60^{o}C in an atmosphere circulated over silica gel and soda lime. The samples were then placed in stainless steel test cups containing a saturated solution of potassium nitrate and sealed in-situ with molten paraffin wax. The air gap between the base of the samples and the solution was 12 mm (±2 mm). The KNO_3 solution generates a relative humidity within the air gap of 93.2% at 20^{o}C. The samples were then placed in a fan-assisted storage chamber maintained at 20^{o}C (±2^{o}C) and 50% RH (±5%) and weighed every 24 hours until steady state vapour transmission was achieved. The reported values represent the mean of three individual samples.

2.3.5 Mercury Intrusion Porosimetry (MIP)

The pore size distributions of the mortars were determined using MIP at an age of 91 days (air cured). The measurements were carried out with a Micromeritics 9400 series instrument over a range of pressures between 3.9×10^{-3} and 410 MPa. Assuming a contact angle of 140^{o} and a mercury surface tension of 484×10^{-3} N/m, pore diameters ranging from 375 μm to 3.8×10^{-3} μm were characterized. Specimens with an approximate volume of 2 cm^3 were cut from the mortar prisms using a low speed saw. Prior to analysis, the samples were dried to constant weight at $60^{o}C$ in an atmosphere circulated over silica gel and soda lime.

3. Properties of "fresh" mortars

3.1 Pore structure

Values of the total open porosity and dry bulk density are shown in Table 3. The general trend observed for all mortars is a slight decrease in porosity, allied with an increase in bulk density, as the curing time was extended, and is more marked in the M1 mortars. The M5 series of mortars show higher density and lower porosity than the M1 series, as would be expected from the higher ggbs/CH ratio. The difference is more marked in mortars produced using either quicklime C1 or the CL90.

Mortar	Open Porosity (%)			Dry Bulk Density (kg/m^3)		
	28 d	91 d	365 d	28 d	91 d	365 d
M1-1	32.2	31.6	31.0	1757	1781	1791
	32.7	*31.8*	*31.4*	*1689*	*1717*	*1729*
M1-2	31.8	31.4	31.0	1769	1794	1799
	32.9	*31.7*	*31.5*	*1684*	*1719*	*1735*
M1-3	32.1	31.4	30.8	1761	1795	1797
	32.8	*31.9*	*31.4*	*1689*	*1714*	*1737*
M1-4	33.5	32.8	32.3	1720	1749	1767
	34.2	*33.1*	*32.6*	*1660*	*1700*	*1709*
M1-5	34.9	34.2	33.7	1693	1686	1701
	35.1	*34.2*	*33.6*	*1651*	*1679*	*1700*
M5-1	29.3	29.3	29.1	1767	1805	1805
	29.9	*28.5*	*27.8*	*1733*	*1746*	*1779*
M5-4	29.4	29.4	29.1	1755	1777	1784
	30.1	*29.1*	*27.7*	*1729*	*1741*	*1775*
M5-5	29.5	29.8	29.3	1745	1759	1767
	30.6	*29.0*	*28.2*	*1721*	*1733*	*1766*

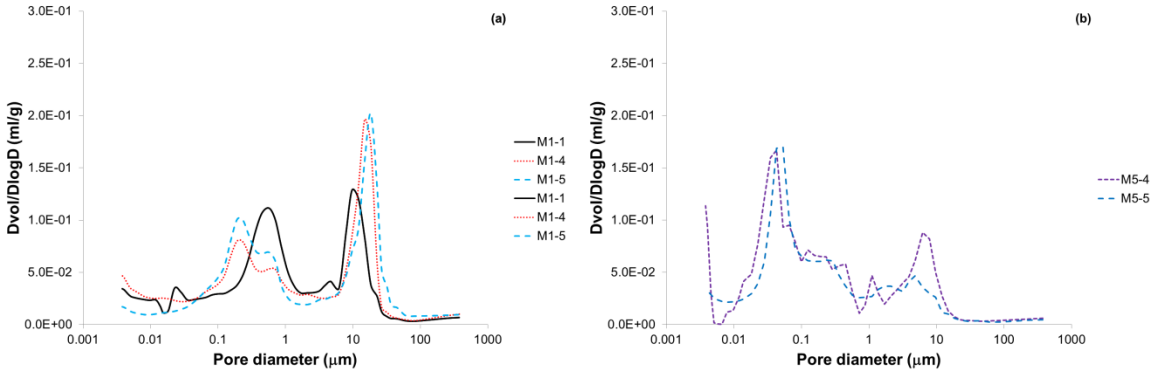
Table 3: Porosity and density of mortars at ages of 28, 91 and 365 days (air curing in Normal font, water curing in Italics).

Within the M1 range of mortars M1-1, M1-2 and M1-3 possess the same porosity at a given age and curing condition; all these mortars were dried with the same quicklime E, the only difference being in the process parameters of stoichiometric ratio and mixing time. The influence of lime type may be seen in the higher porosities of M1-4 (quicklime

C1) and M1-5 (CL90). However, any influence of lime type in the M5 mortars (M5-1, M5-4 and M5-5) is reduced to a minimum, probably as a function of the reduced lime content of these mortars.

The influence of curing regime is small for the M1 mortars although water curing yields the slightly higher porosity at all ages. In contrast, water-cured M5 show higher porosity at 28 days but lower porosity at 365 days, presumably reflecting additional hydraulic activity at later ages.

The pore size distributions of the mortars (91 days of air curing) are shown in Figure 2. The data highlight significant differences in the pore size distributions between the M1 (Fig 2a) and M5 (Fig 2b) mortar series. The lime-rich M1 series contain a large proportion of pores in 2 distinct zones, i.e. 10 – 18 μm and 0.2 – 0.6 μm. Mortars M1-4 and M1-5 exhibit similar pore structures; both mortars are based upon lime from the same source. Mortar M1-1 shows a slightly lower threshold pore diameter and a lower volume of the coarsest pores which accompanies the lowest open porosity (Table 3). In contrast, for the M5 mortars, the bulk of the porosity is made up of pores in the region of 0.05 μm diameter. This is a function of the higher ggbs/CH ratio and lower w/b ratio of the M5 series. As with the M1 mortars, the pore distributions of M5 mortars using quicklime C1 and CL90 are similar.



269 Fig 2: Pore size distribution of (a) M1 and (b) M5 mortars after 91 days of air-curing.

270

271 A similar pattern of small differences in open porosity accompanied by marked
272 differences in pore size distribution has been previously reported by Arandigoyen and
273 Alvarez [13] for cement-lime mortars.

274

275 **3.2 Compressive and flexural strength**

276

277 **3.2.1 M1 mortar formulations**

278

279 The data in Figures 3a and 3b show the evolution of flexural and compressive strength
280 of the M1 mortars subjected to air curing. Under flexural load, mixes M1-1 to M1-5 yield
281 similar strengths at 7 days. All mortars show a considerable and similar gain in flexural
282 strength up to 91 days (all significance testing used the student t-test conducted at 95%
283 confidence). Under compression, mortars M1-1 to M1-5 display essentially similar initial
284 strength development up to 28 days. Thereafter, mortars M1-1, M1-2 and M1-3 show a
285 further strength increase up to 91 days with no further change between 91 and 365
286 days. In contrast, mortars M1-4 and M1-5 show no gain in compressive strength after 28
287 days and, consequently, these materials are significantly weaker at the later ages.

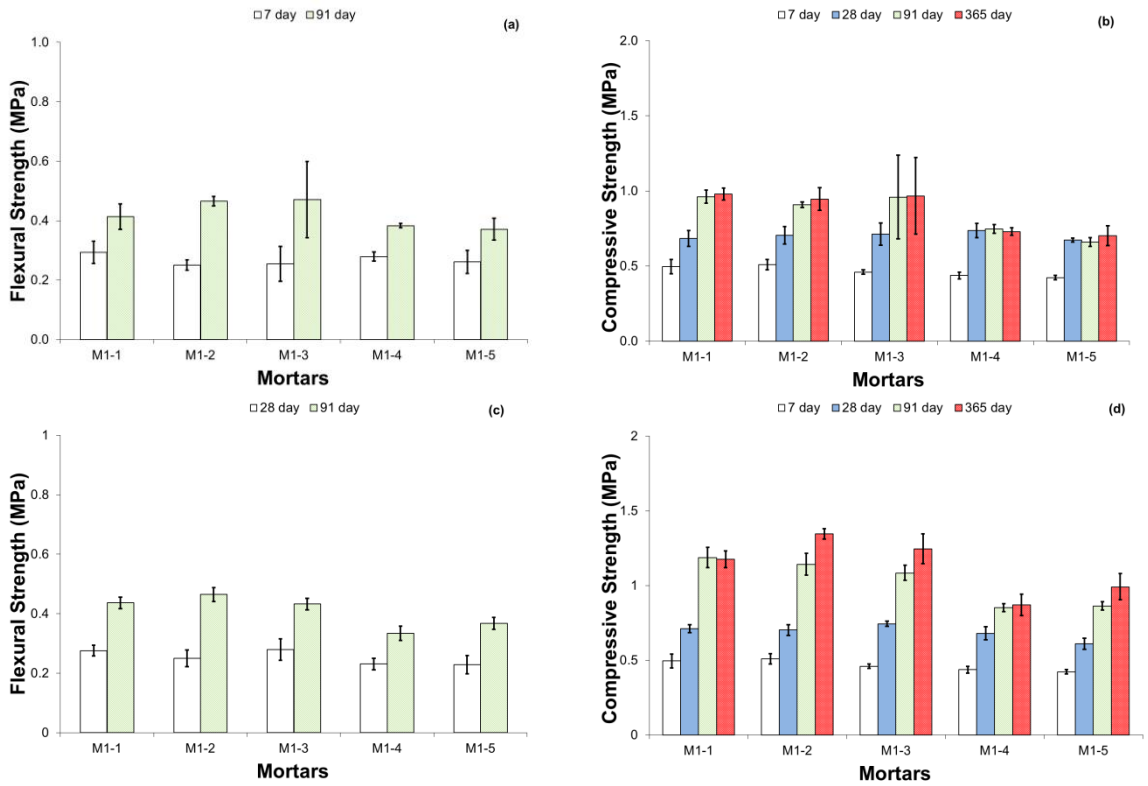


Fig 3: Strengths of M1 mortars – (a) flexural strength air curing, (b) compressive strength air curing, (c) flexural strength water curing, (d) compressive strength water curing. Error bars represent ±1 Standard Deviation.

Figures 3c and 3d display the influence of water curing on the strength development of the mortars M1-1 to M1-5. In flexure, mortars M1-1, M1-2 and M1-3 exhibit similar strengths at 91 days with mortars M1-4 and M1-5 also being similar but weaker than the other mortars. With the exception of M1-4, the strength of all mortars is similar at 91 days for both curing conditions. Up to an age of 28 days, all mortars show similar compressive strengths and there is little difference in the compressive strengths of samples subjected to the different curing conditions. However, at ages of 91 and 365 days water curing generally generates higher strength (typically 20 – 40%) reflecting the extra potential for hydraulic reactions in the lime-ggbs system. The exception is mortar M1-3 which exhibited a high variability of strength under air curing at these ages, so

influencing the statistical comparison; it is not believed that the apparent difference exhibited by this mortar formulation is genuine.

3.2.2 M5 mortar formulations

The data in Figures 4a and 4b show the evolution of flexural and compressive strength of the M5 mortars. There is little influence of lime type in the M5 mortars. As expected the highest strengths are registered by water cured samples such that by an age of 1 year a strength enhancement in the range 130 – 140% is observed. Whilst the latter curing yields continuous strength increase up to an age of 1 year, air cured samples indicate a maximum strength at either 28 or 91 days with a subsequent reduction at an age of 1 year; this was not observed in the M1 mortars.

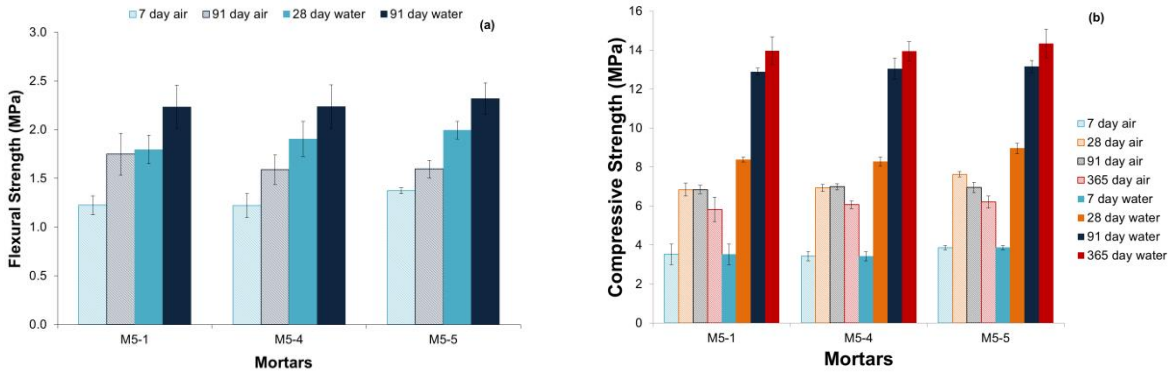


Fig 4: Strengths of M5 mortars – (a) flexural strength, (b) compressive strength. Error bars represent ±1 Standard Deviation.

The influence of curing regime on long term development of alkali-activated slag concrete (sodium silicate and calcium hydroxide activation) [14] and of calcium hydroxide activated slag mortars [15] has been previously observed. The strength reduction was explained by the high shrinkage and micro-cracking associated with this binder system. It may be observed that the absence of strength reduction is seen in the

lime-rich M1 mortars and, whilst any correlation is purely speculative, the strength reduction in the M5 and calcium hydroxide activated slag mortars [15] occurs in mortars with a larger hydraulic component and less lime.

Both compressive and flexural strengths have been measured for all mortars and curing conditions at 2 ages. The ratio of compressive:flexural strength as a function of compressive strength is shown in Figure 5. As expected the ratio increases as the compressive strength increases reflecting a more brittle material. There is a certain degree of scatter, particularly at lower strengths; a correlation coefficient of 0.872 is achieved.

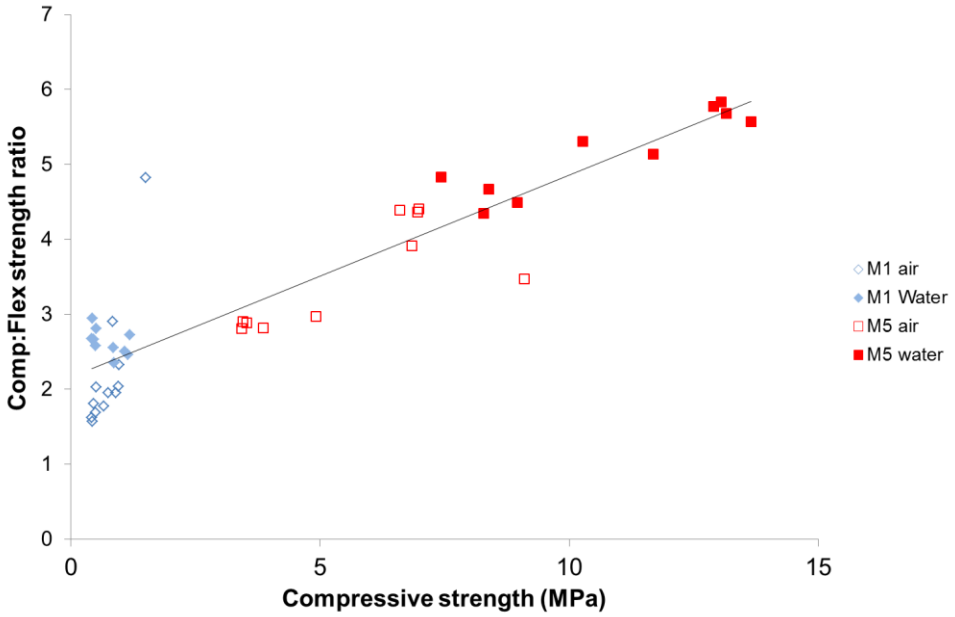


Fig 5: Ratio of compressive:flexural strength as a function of compressive strength for M1 and M5 mortars.

Both M1 and M5 mortars generally achieve their strength designation at an age between 28 and 91 days of water curing although it is likely the time is towards the lower end of this range for the M5 mortars.

3.3 Moisture transport

The specification of mortars is dependent upon their end-use. In the case of restoration works on historic buildings their breathability is an important property. However, this is a topic with differing philosophical approaches adopted by different restorers. One approach, as codified in BS EN 998-1:2010 [16], may be summarized as one in which the movement of water is to be minimised where-as the movement of water vapour is to be encouraged. This approach frequently requires the use of hydrophobic treatments within the mortar system. Another viewpoint is that this approach only transfers the original problems to other areas of the structure which have not been treated with such a mortar [e.g. 17, 18]. It is argued that since water transport is much quicker than vapour transport both parameters are important.

In the current work, breathability was assessed by sorptivity and water vapour permeability (WVP) measurements following air curing and the results are presented in Figures 6 and 7 respectively.

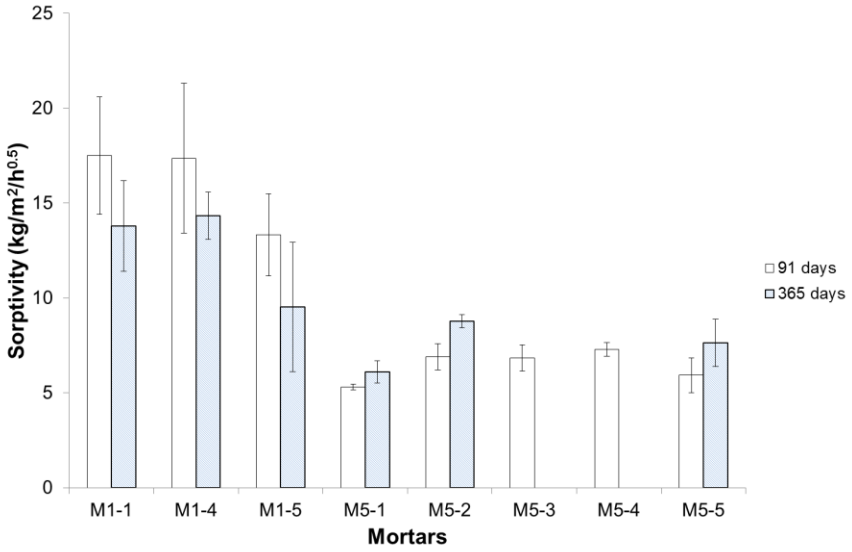


Fig 6: Sorptivity of M1 and M5 mortars.

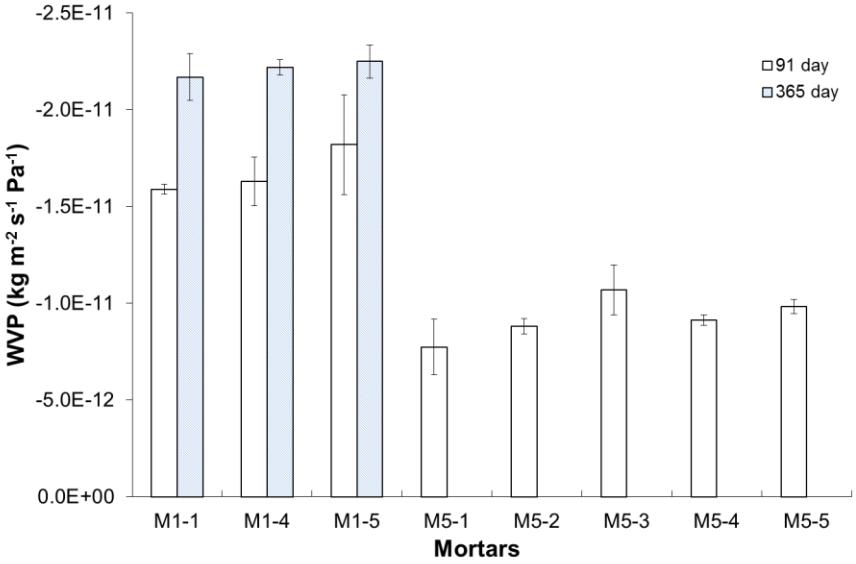


Fig 7: WVP of M1 and M5 mortars.

It is apparent that the sorptivity and WVP of the M5 mortars is less than that of the M1 mortars reflecting their finer pore structure. Although the average values suggest that all mortars show a variation in sorptivity with age, the variability within each data set is such that there is no statistical difference between mortars. By way of contrast, the WVP of the M1 mortars generally increases with age although that of the M1-5 mortar only marginally fails the significance test. There is no influence of lime source on the sorptivity or WVP of the M1 mortars at ages either 91 or 365 days unlike that observed for compressive strength, nor on the sorptivity or WVP of the M5 mortars at 91 days which is the only test age with data for all of this group of mortars.

Both M1 and M5 mortars exceed a commonly accepted value of 4 $Kg/m^2/hr^{0.5}$ for the sorptivity, or Water Absorption Coefficient, for restoration mortars for renders [4]. Obviously, neither mortar type complies with the low water absorption criterion of BS EN 998-1 although the M1 mortars do meet the requirement for WVP (μ >15).

The lower “permeability” of the M5 mortars is reflected in their reduced rate of carbonation as measured by depths of complete carbonation using the phenolphthalein test (Table 4).

Mortar	28d	91d	365d
M1	2 - 3	9 - 10	Complete
M5	1 - 3	6 - 7	13 - 15

Table 4: Carbonation depths (mm) of M1 and M5 mortars

3.4 Discussion

An influence of lime type has been observed in strength performance but not in moisture transport. It is possible that this may be a function of the greater variability within the latter determinations and had a larger number of samples been tested at each stage a significant difference might have been obtained. The following discussion will focus on the strength data.

The similar performance of the M1-1, M1-2 and M1-3 mortars is to be expected as the materials are of essentially identical composition with the key parameters of lime source and ggbs/CH & w/b ratios being common. The data provides confirmation that manipulation of the lime drying process (quicklime stoichiometric ratio and mixing time) allows the production of identical mortars from quarried aggregates containing different amounts of free water. Likewise, the performance of mortars M1-4 and M1-5 may be similarly explained; despite M1-4 being a lime-dried mortar and M1-5 comprising a commercial CL90, the source of quicklime in both cases is the same.

It was previously shown that lime E yielded higher long term strengths than lime C1 in the M1 mortars but not in the M5 mortars (sections 3.2.1 and 3.2.2). The differences in BET surface area (Table 1) do not account for the different performances; however, the greater strength of the mortars produced with lime E may reflect the higher $CaCO_3$ content of the slaked lime. In this case, the lime-rich M1 mortars contain a larger quantity of $CaCO_3$ than do the M5 mortars. Studies of the use of $CaCO_3$ as an addition to Portland cement [19] show that fine $CaCO_3$ can be both a reactive component as well as a filler. Additional hydration within the AF_m phases is observed at low carbonate additions and is accompanied by reduced porosity. A study of the inclusion of metakaolin and limestone in Portland cement mortars has shown that the carbonate reacts with the metakaolin to produce AF_m phases [20]. The identification of AF_m in alkali-activated cements is uncertain and may be depend upon detection technique and the particular alkali utilized [e.g. 21, 22]. Further, whilst the addition of 1-3% fine calcite has been found to yield a small increase in the early age strength of an alkali-activated cement, no or even a negative impact occurred at later ages [19]. Little modern research has been undertaken on the lime-ggbs system and certainly not in the low slag contents currently being considered and further fundamental research is required to examine the role of $CaCO_3$ within this system.

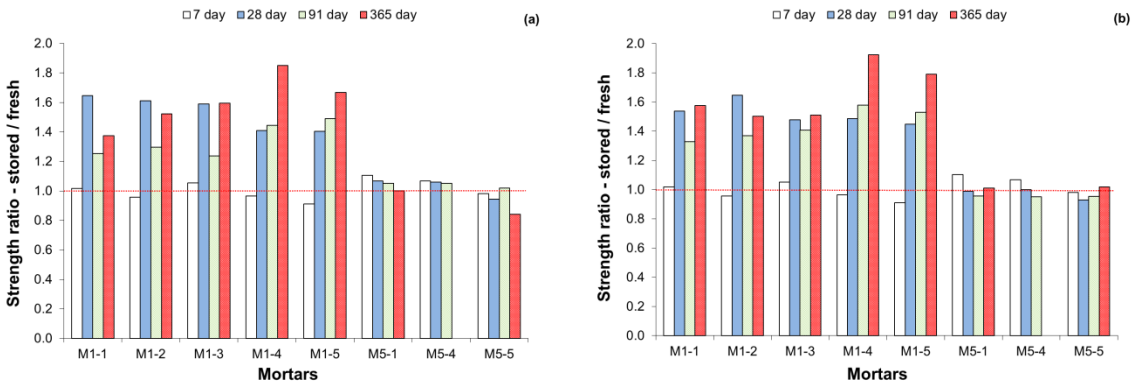
In contrast to the M1 mortars, the strength development of M5-1, M5-4 and M5-5 mortars appears unaffected by the different limes used in their manufacture. The reasons for this difference are unclear but the discrepancy indicates that the properties of the lime are of greater significance in mortars with higher lime content. It seems reasonable to assume that at low ggbs/lime ratios, the development of the mortar microstructure would be more significantly influenced by changes in lime properties than

in mortars where the lime content is much reduced. In cement-lime-sand mortar systems, Sebaibi *et al.* [24] stated that 'it is necessary to have a high lime substitution percentage to influence the microstructure of the mortar'; however, the PC/lime ratios used in their mortar formulations were far higher than the ggbs/lime ratios employed in the current work.

4 The effect of storage on lime-dried mortar mixes

4.1 Optimally dried mortars

The data shows that the performance of lime-dried mortars is very similar to those produced using commercial CL90 hydrated lime. However, one of the essential requirements of the lime-drying process is the ability to produce mortars that can be stored in silos or bags without significant degradation prior to use. The lime-drying process is unlikely to remove water absorbed within the pores of the aggregate unless excessive heat is generated during the slaking process. As a pre-cursor to work on lime-dried mortars a range of mortars was produced using NHL 3.5 as the binder phase in which the sand was pre-prepared at various moisture states between oven dry and saturated surface dry. They were blended in the volume ratio 2.25:1 and stored for 8 weeks prior to mixing into mortars of common flow. There was no statistical difference (95% confidence limits) between mortar strengths at ages up to 2 years indicating no influence of absorbed water on mortar performance. Hence, a series of lime-dried mortars was produced following a 10-week storage (see section 2.2.2) in which the expectation was that any influence of storage would be a function of free water rather than absorbed water.



447 Fig 8: Relative strengths of stored and fresh mortars; (a) air cured, (b) water cured.

448

449 Figure 8 shows the relative compressive strength of the M1 and M5 series of "stored"
450 mortars (both water and air-cured) in comparison to the "freshly" prepared materials
451 described in section 3.2. It is apparent that there is a difference in performance between
452 the M1 and M5 mortars. The M5 "stored" mortars are generally similar to their "fresh"
453 counterparts at all ages except 365 days where the air-cured "stored" mortars lose
454 strength at a more rapid rate than the "fresh" mortars (see Fig 8a). At 365 days the
455 "stored" mortars are some 77% of the 91 day strengths in contrast to 87% for the "fresh"
456 mortars. In contrast, the strengths of the M1 "stored" mortars are considerably greater
457 than those of the fresh materials at ages beyond 7 days under both air and water curing.
458 The strength increases are accompanied by an increase in bulk density and a decrease
459 in the porosity in the stored mixes (Table 5).

Mortar	Open porosity (%)		Density (kg/m^3)	
	Fresh	Stored	Fresh	Stored
M1-1	31.6	30.6	1781	1809
M1-2	31.4	30.6	1794	1808
M1-3	31.4	30.2	1795	1818
M1-4	32.8	30.9	1749	1803
M1-5	34.2	32.6	1686	1766

Table 5: Influence of storage on M1 mortars

Only a very limited study was undertaken on M1 mortars (M1-1, M1-4 & M1-5) to examine the influence of storage upon the moisture transport properties at an age of 91 days curing in air. Whilst strength was affected by storage there is no significant influence on moisture transport.

The precise reason for the observed differences between the M5 and M1 mortar series is unclear but appears to be related to changes in the water demand of the plastic mortars following storage. Table 6 shows the w/b ratios required to achieve the specified flow values during mortar manufacture. For the M1 series, the data show a marked reduction in water demand after storage whereas comparatively little change is observed for the M5 series. It seems reasonable to assume that the reduction in water demand is a result of changes in the properties of the calcium hydroxide during storage. These changes were discussed in detail elsewhere [1] and include decreases in surface area and possibly a small increase in the quantity of calcium carbonate within the lime; see Tables 8 and 9 of ref [1]. For a given lime, it seems likely that a reduction in surface area would lead to a lower water demand in the plastic mortars and, as discussed previously, the presence of calcium carbonate leads to enhanced strength in lime mortars.

479 Furthermore, the high proportion of lime in the M1 mixes suggests that these materials
480 would be more sensitive to changes in lime composition than the slag-rich M5 series.
481 Despite the increasing use of pre-mixed lime mortars in UK construction practice, there
482 is no evidence within the literature of research to determine potential changes in the
483 properties of these materials following extended periods of silo storage. In the current
484 paper, changes were not confined to mortars prepared with the lime-drying technique
485 (see mortar M1-5) and it seems reasonable to assume that other lime rich mortars may
486 also be subject to the same phenomenon. Hence, further research is recommended in
487 this area.

Mortar	w/b ratio		Change
	"Fresh"	"Stored"	(%)
M1-1	1.35	1.30	3.7
M1-2	1.36	1.31	3.7
M1-3	1.36	1.29	5.1
M1-4	1.37	1.31	4.4
M1-5	1.41	1.35	4.3
M5-1	1.03	1.01	1.9
M5-2	1.01	1.00	1.0
M5-3	0.99	0.98	1.0

488 Table 6: Influence of storage on w/b ratio to achieve common flow.
489

490 **4.2 Non-optimally dried mortars**

491

492 Three regions of drying have been previously defined [1]. Optimal drying is considered to
493 occur when the measured evaporation during the drying process equals the balance of

494 the original free moisture and that removed by chemical combination after accounting for
495 the stoichiometric ratio of quicklime used and its purity. In contrast, sub-optimal drying
496 does not remove sufficient water and super-optimal drying removes too much water
497 leaving a residual amount of free lime - see ref [1] for a fuller discussion. The
498 assumption was that sub-optimal drying would lead to degradation during storage whilst
499 super-optimal drying would yield an unsound binder. The latter has previously been
500 confirmed for storage periods of up to 4 weeks [1].
501
502 A series of 10 mortars was produced based upon the optimal processing criteria for the
503 M1-1 mortar. By reducing the stoichiometric ratio of quicklime sub-optimal mortars were
504 produced and by increasing it super-optimal mortars resulted. After lime-drying half the
505 mortar was used to produce “fresh” mortars whilst the remainder was stored in sealed
506 boxes for 10 weeks to produce “stored” mortars. All mortars were produced to a
507 common flow of 170 mm and subject to both air and water curing. Table 7 shows the key
508 data of stoichiometric ratio and the stoichiometric variation from optimal conditions for
509 each mortar together with the w/b ratio to achieve the desired flow. It can be seen that
510 the optimally dried mortar has the highest water demand of all mortars; the reason for
511 which is not immediately apparent.

Stoichiometric ratio	Stoichiometric variation	w/b	
(%)	(%)	"Fresh"	"Stored"
50.0	-9	1.18	1.17
54.9	-4	1.21	1.25
55.9	-3	1.21	1.26
56.9	-2	1.24	1.24
57.9	-1	1.28	1.23
58.9	Optimal	1.35	1.31
59.9	+1	1.26	1.22
60.9	+2	1.28	1.22
61.9	+3	1.28	1.20
67.0	+8	1.26	1.18

Table 7: Sub-optimal, optimal and super-optimal mortars

The water demand of the "stored" optimal and super-optimal mortars is less than their "fresh" counterparts as previously observed in section 4.1. This might be explained by the presence of unslaked lime in the "fresh" mortars placing a demand for water to slake the quicklime during mortar production whilst this was air-slaked during the storage period. In contrast, the trend for the sub-optimal mortars is less clear but, in general, appears to be either unaffected or shows an increase in water demand which may be a reflection of the generation of increased surface area by hydration of ggbs during storage.

It is clear from the raw data that the use of super-optimal drying has not been detrimental to strength development under either storage period or curing condition. This

would support the supposition that any free lime remaining after lime-drying has been converted to slaked lime during the storage period or the mortar mixing process. The relative performance of each mortar, expressed as the ratio of the compressive strength of the “stored” / “fresh” mortars at ages between 7 – 365 days is shown in Figure 9. Under both curing conditions both the optimal and super-optimal mortars display a strength enhancement following the 10 week storage period; the super-optimal +1% mortar may be an anomaly. Whilst the sub-optimal -1% and -2% mortars subject to air curing also show strength enhancement at all ages, the water cured samples register a reduction in relative strength with age suggesting that the longer term hydration of the ggbs may have been compromised during storage. The sub-optimal -3% mortar appears to present a threshold in behaviour in that whilst storage has not affected the strength neither is strength enhancement observed. As the stored mortars become wetter (-4% and -9% variation) it is clear that they have degraded to the extent that the “stored” mortars are substantially weaker at all ages, more so following water curing than air curing.

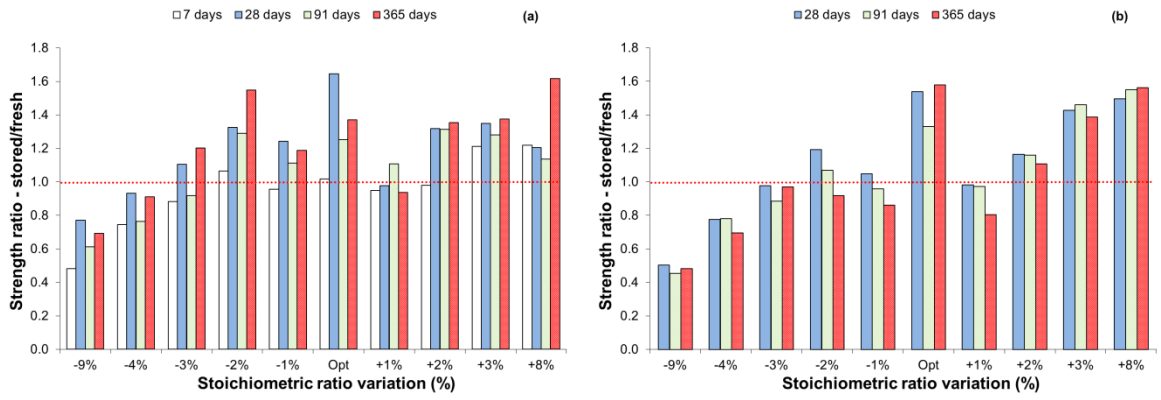


Fig 9: Influence of varying the quicklime addition on relative strength of “fresh” and “stored” mortars subject to (a) air curing and (b) water curing.

This data suggest that in the practical implementation of the lime-drying process there is a narrow range of quicklime addition in which the quality of mortar is maintained whilst also minimizing the financial cost of using excessive quicklime.

Part B – Roman cement mortars

5 Materials and Methods

A Roman cement developed during the EU funded ROCARE project from marls sourced in Gartenau, Austria, and a 0 - 4 mm carbonate sand were used. The cement was manufactured by The Institute of Ceramics and Building Materials (MBM) in Krakow, Poland; details may be found elsewhere [5]. Two mixes have been de-activated with 7% de-activation water and 30 min storage prior to the formulation of the final mortar. The deactivation water, expressed as a percentage of the cement weight, is first added to the oven dry sand and mixed for 2 min at 62 rpm (a Hobart mixer was used for all mortar production). Subsequently, the cement was added to the wet sand and the whole mixed for a further 2 minutes at 62 rpm before being stored in an airtight box. At the end of the 30 minute de-activation storage time, the first mix was oven dried for 24 hours at 105°C in an atmosphere circulated over silica gel and soda lime while the second mix was lime-dried by means of the addition of a pre-determined amount of quicklime. The moisture content of the DARC mortar was determined as 0.63%. Based on previous experience the lime content was specified as 76% stoichiometric ratio with a 5 minute mixing time. Both mixes were then stored in airtight boxes for 4 weeks prior to formation into mortars. A third mix (control mix) was prepared and stored for the same period of time with no drying carried out.

569 At the end of the 4 week storage period, mortars were manufactured at a sand:cement
570 ratio of 2.5:1 by volume and constant w/c ratio of 0.81. The mortars were produced by
571 mixing the products of the previous drying processes in a Hobart mixer with water for 30
572 s at 62 rpm. At this time the mixer was stopped for 30 s during which the mortar
573 adhering to the wall and bottom part of the bowl was removed by means of a rubber
574 scraper and placed in the middle of the bowl. The mixing was then continued at 125 rpm
575 for 8 minutes.
576
577 Flow and workable life were measured according to BS EN 1015-3:1999 [7] and BS EN
578 1015-9:1999 [25] respectively. Samples for strength determination were cast in 40 × 40
579 × 160 mm steel moulds in two layers and vibration compacted before being covered with
580 a polythene sheet. Mortar beams were de-moulded after 24 h and cured in water at 20°C
581 prior to testing at 28 days.
582
583 Differential Thermo-gravimetric (DTG) tests (a heating rate of 5°C per minute to 150°C
584 under a nitrogen atmosphere) were also performed on the oven dried mix and on the
585 control mix immediately after the end of the 30 minute storage period.
586

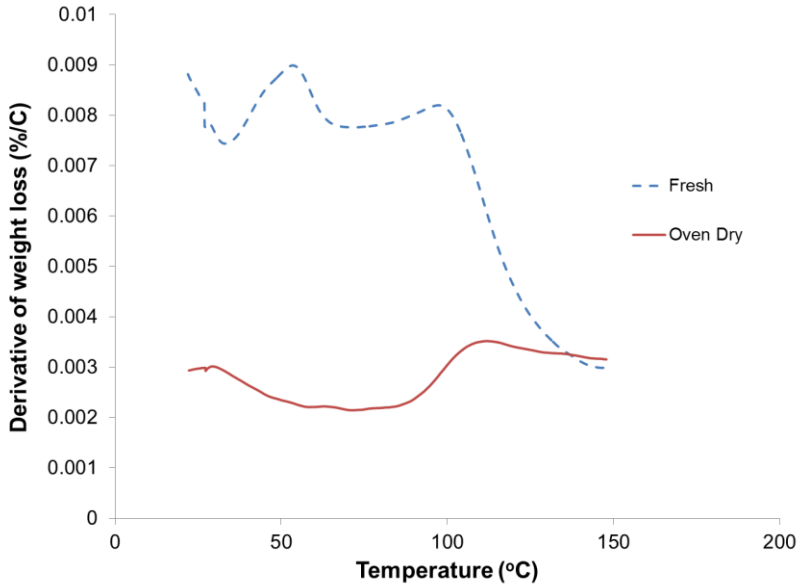
6 Mortar properties

Mortar	Flow (mm)	Work Life (min)	Comp Strength (MPa)	Flex Strength (MPa)
Control	170	>141	9.84	2.98
Oven Dried	155	~50	11.8	3.51
Lime Dried	140	~47	13.2	3.92

Table 8: Properties of Roman cement mortars

In Table 8 it is shown that both the oven dried and the lime-dried mortars exhibit a lower fluidity than that of the control mortar, with the lowest flow being observed for the lime-dried mortar. This means that these mortars will require higher amounts of water to achieve the desired workability. Whilst the higher water demand of the lime dried mortar can be explained by the presence in this mix of calcium hydroxide formed in the reaction of quicklime with de-activation water, it was not expected that the oven dried mix would have a higher water demand than that of the control mix. In fact, in a previous study [5] it was shown that during storage of a DARC mortar (i.e. the control mortar) the water demand for the subsequent wet mortar was increased as the storage period was prolonged. Whilst all 3 “dry” mortars in the current study were stored for 4 weeks, the oven-dried mortar was conditioned to a lower free moisture content such it might have been expected to perform similarly to a DARC mortar that had been subject to a 30 min only storage period, i.e. a lower water demand or higher fluidity than that of the control mortar. A possible explanation of this data is that the oven-drying causes the de-hydroxylation of the AF_m phases. In order to verify this hypothesis, DTG tests were undertaken on the control mix (fresh) and on the oven-dried (OD) mix and results are

607 shown in Figure 10. It is apparent that the use of oven drying has removed water from
608 the system which may be accounted for by both the loss of evaporable water [26] and
609 partial de-hydroxylation of the AF_m phases [27]. In this case, the reverse process (re-
610 hydroxylation) might take place during the early life of the constituted mortar and
611 account for the increased demand for mixing water.



612

613 Fig 10: DTG curves of control (fresh) mix and oven dried (OD) mix (5°C per minute to
614 150°C).

615

616 Table 8 also shows that the workable life of the mortar produced with the control mix is
617 at least 3 times longer than that produced with the oven dried and with the lime-dried
618 mix. This can be in part attributed to the higher flow of the control mix mortar and in part
619 to the longer storage under conditions of free moisture experienced by this mix, which
620 has been shown to increase workable life [5]. Further study including the production and
621 testing of mortars at constant flow is necessary to better understand the effect of the
622 drying processes on workable life. Both compressive and flexural strength (28 days) are

623 higher for the dried mix mortars than for the control mix mortar, with the highest strength
624 observed for the lime-dried mix mortar.
625
626 It is not possible to undertake a strict comparison between the degradation observed in
627 the stored DARC mortars (see Figs. 13 & 14, ref 5) and the comparative performance of
628 the various mortars reported here-in since the sand:cement and w/c ratios are different
629 in the two studies. However, the conditioning of the current control mix equates to that of
630 the DARC mortar stored for 4 weeks in [5]. Using these mortars as the basis for
631 comparison the strength of the lime-dried mortar is 134% of the control mortar whilst the
632 strength of the 30 minute stored DARC mortar is 152% of that of the 4 week stored
633 DARC mortar [5]. Thus, the lime-drying has largely mitigated the degradation attributed
634 to the effects of free moisture present during prolonged storage of the DARC mortars; all
635 free moisture would not have been immediately removed by the addition of quicklime so
636 some residual degradation would be expected.
637
638 Experience suggests that once the water content of the lime-dried mortar is adjusted to
639 yield the desired flow, the strength would be similar to that of the control mix and the
640 workable life extended into the target range of 1 – 2 hours; additional work is required to
641 prove this assumption.
642
643 **7 Conclusions**
644
645 From the results described in Parts A and B of this paper it can be concluded that:
646
647 • The lime-drying process has been successfully employed to produce mortars of M1
648 and M5 classification using ggbs as the hydraulic component.

- By careful manipulation of the amount of quicklime added (stoichiometric ratio) and the length of the mixing time during the lime-drying process it is possible to produce identical mortars from sands with differing water contents.
- The composition of different quicklimes may have a significant influence on the strength of hardened mortars prepared from the lime drying process, particularly in lime-rich blends with low ggbs/CH.
- The strength of the lime-slag mortars produced by the lime-drying process are very similar to those of mortars produced using equivalent commercial hydrated lime.
- The lime dried materials may be silo-stored prior to use without changes to the mortar classification.
- The work has highlighted a requirement for further research to examine the effects of silo storage on the subsequent water demand and strength of lime mortars containing a high proportion of lime.
- Lime-drying overcomes strength degradation previously observed in Roman cement mortars retarded by a pre-hydration technique and subsequently stored.

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675

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